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3-(3-Methylphenyl)-2-thioxo-1,3-thiazolidin-4-one

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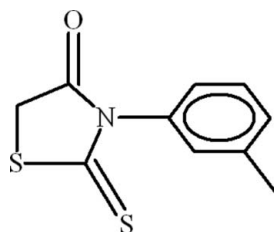
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.032; wR factor = 0.087; data-to-parameter ratio = 20.7.

In the title compound, $\text{C}_{10}\text{H}_9\text{NOS}_2$, the dihedral angle between the rhodanine (2-thioxo-1,3-thiazolidin-4-one) and 3-methylphenyl rings is 83.30 (3)°. The H atoms of the methyl group are disordered over two set of sites with an occupancy ratio of 0.58 (3):0.42 (3). In the crystal, the molecules interact by way of $\text{C}-\text{H}\cdots\pi$ and $\text{C}=\text{O}\cdots\pi$ interactions.

Related literature

For related structures, see: Shahwar *et al.* (2009a,b,c,d,e).

Experimental

Crystal data

 $\text{C}_{10}\text{H}_9\text{NOS}_2$ $M_r = 223.3$ Monoclinic, $P2_1/c$ $a = 8.0775$ (3) Å $b = 6.4058$ (2) Å $c = 21.4715$ (7) Å $\beta = 106.068$ (2)° $V = 1067.59$ (6) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.46$ mm⁻¹ $T = 296$ K $0.32 \times 0.24 \times 0.22$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 2005)

 $T_{\min} = 0.849$, $T_{\max} = 0.897$

11841 measured reflections

2666 independent reflections

2116 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.026$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.032$ $wR(F^2) = 0.087$ $S = 1.03$

2666 reflections

129 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.27$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C8}-\text{H8B}\cdots\text{Cg2}^{\text{i}}$	0.97	2.59	3.5219 (17)	162
$\text{C7}-\text{O1}\cdots\text{Cg1}^{\text{i}}$	1.20 (1)	2.94 (1)	4.1070 (16)	164 (1)

Symmetry code: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$. Cg1 and Cg2 are the centroids of the S1/C8/C7/N1/C9 and C1-C6 rings, respectively.

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997) and PLATON (Spek, 2009); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5208).

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supporting information

Acta Cryst. (2009). E65, o3016 [doi:10.1107/S1600536809045863]

3-(3-Methylphenyl)-2-thioxo-1,3-thiazolidin-4-one

Durre Shahwar, M. Nawaz Tahir, Asma Yasmeen, Naeem Ahmad and Muhammad Akmal Khan

S1. Comment

We have reported the synthesis and crystal structures of various rhodanine derivatives such as (II) (5*Z*)-5-(2-Hydroxybenzylidene)-3-phenyl-2-thioxo-1,3-thiazolidin-4-one (Shahwar *et al.*, 2009*a*), (III) (5*E*)-5-(4-Hydroxy-3-methoxybenzylidene)-2-thioxo-1,3-thiazolidin-4-one methanol monosolvate (Shahwar *et al.*, 2009*b*), (IV) (5*Z*)-5-(2-Hydroxybenzylidene)-2-thioxo-1,3-thiazolidin-4-one methanol hemisolvate (Shahwar *et al.*, 2009*c*), (V) 3-(2-Methylphenyl)-2-thioxo-1,3-thiazolidin-4-one (Shahwar *et al.*, 2009*d*) and (VI) 3-Cyclohexyl-2-thioxo-1,3-thiazolidin-4-one (Shahwar *et al.*, 2009*e*). The purpose of synthesis of different rhodanine derivatives is to study the biological activities. The title compound (I, Fig. 1) is being reported in this context.

In (I), the 3-methylphenyl A (C1–C6/C10) and the rhodanine group B (N1/C7/C8/S1/C9/O1/S2) are planar with maximum r. m. s. deviations of 0.0068 and 0.0171 Å respectively, from their mean square planes. The dihedral angle between A/B is 83.30 (3)°. The H-atoms of the methyl moiety are disordered over two set of sites with occupancy ratio of 0.58 (3):0.42 (3) in the monomers. There exist C–H···π and C=O···π interactions (Table 1) which stabilize the molecules.

S2. Experimental

The title compound was prepared by a three step reaction procedure. In the first step *meta* toluidine aniline (10.7 g, 0.1 mol) and triethylamine (50.5 g, 0.5 mol) were stirred in ethanol (20 ml) followed by dropwise addition of CS₂ (15.2 g, 0.2 mol) while keeping the flask in an ice bath. The precipitate obtained were filtered off and washed with diethyl ether.

In second step, a solution of sodium chloroacetate (11.6 g, 0.1 mol) and chloroacetic acid (18.9 g, 0.2 mol) was prepared in 50 ml distilled water. To this solution the precipitates obtained in first step were added gradually and stirred at 273 K. This mixture was stirred until it turned light yellow.

In third step the yellow mixture was mixed in 140 ml hot (363–368 K) hydrochloric acid (6 N) and stirred for five minutes to obtain colorless crystalline precipitates. These precipitates were recrystallized in chloroform to get light yellow prisms of (I).

S3. Refinement

The H-atoms were positioned geometrically (C–H = 0.93–0.97 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

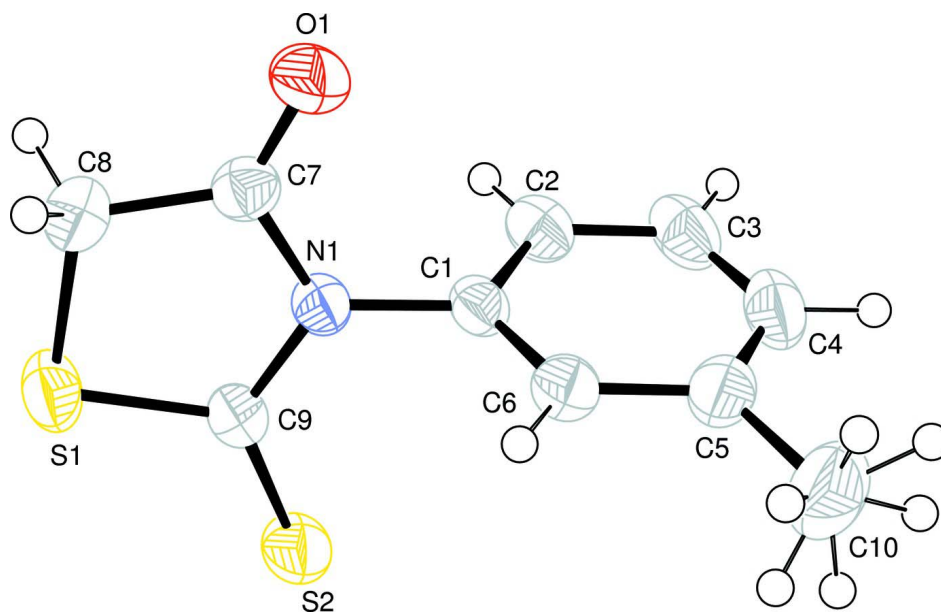


Figure 1

View of (I) with displacement ellipsoids drawn at the 50% probability level.

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Crystal data

$C_{10}H_9NOS_2$

$M_r = 223.3$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 8.0775$ (3) Å

$b = 6.4058$ (2) Å

$c = 21.4715$ (7) Å

$\beta = 106.068$ (2)°

$V = 1067.59$ (6) Å³

$Z = 4$

$F(000) = 464$

$D_x = 1.389$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2666 reflections

$\theta = 2.6$ – 28.3 °

$\mu = 0.46$ mm⁻¹

$T = 296$ K

Prism, light yellow

$0.32 \times 0.24 \times 0.22$ mm

Data collection

Bruker Kappa APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 7.40 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2005)

$T_{\min} = 0.849$, $T_{\max} = 0.897$

11841 measured reflections

2666 independent reflections

2116 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.026$

$\theta_{\max} = 28.3$ °, $\theta_{\min} = 2.6$ °

$h = -10 \rightarrow 10$

$k = -8 \rightarrow 8$

$l = -28 \rightarrow 28$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.087$

$S = 1.03$

2666 reflections

129 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites
H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0399P)^2 + 0.2635P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
S1	0.90452 (5)	0.42703 (7)	0.26996 (2)	0.0484 (1)	
S2	0.85641 (6)	0.38555 (7)	0.12935 (2)	0.0511 (2)	
O1	0.54228 (16)	0.04248 (19)	0.26950 (6)	0.0537 (4)	
N1	0.67528 (14)	0.18699 (18)	0.19889 (5)	0.0324 (3)	
C1	0.56186 (18)	0.0913 (2)	0.14230 (7)	0.0337 (4)	
C2	0.5937 (2)	-0.1103 (2)	0.12588 (8)	0.0419 (5)	
C3	0.4818 (2)	-0.1972 (3)	0.07148 (8)	0.0501 (6)	
C4	0.3436 (2)	-0.0858 (3)	0.03533 (8)	0.0518 (6)	
C5	0.3103 (2)	0.1156 (3)	0.05186 (7)	0.0461 (5)	
C6	0.42217 (18)	0.2036 (3)	0.10687 (7)	0.0391 (5)	
C7	0.65129 (19)	0.1553 (2)	0.26005 (7)	0.0363 (4)	
C8	0.77847 (19)	0.2774 (3)	0.31088 (7)	0.0410 (5)	
C9	0.80207 (17)	0.3243 (2)	0.19488 (7)	0.0342 (4)	
C10	0.1604 (2)	0.2403 (4)	0.01220 (10)	0.0730 (8)	
H2	0.68758	-0.18517	0.15069	0.0502*	
H3	0.50036	-0.33254	0.05925	0.0602*	
H4	0.27025	-0.14709	-0.00126	0.0621*	
H6	0.40257	0.33792	0.11964	0.0469*	
H8A	0.85188	0.18403	0.34219	0.0492*	
H8B	0.71912	0.36922	0.33352	0.0492*	
H10A	0.06610	0.23026	0.03116	0.1095*	0.58 (3)
H10B	0.12550	0.18649	-0.03121	0.1095*	0.58 (3)
H10C	0.19388	0.38382	0.01132	0.1095*	0.58 (3)
H10D	0.19424	0.31376	-0.02127	0.1095*	0.42 (3)
H10E	0.12470	0.33875	0.03963	0.1095*	0.42 (3)
H10F	0.06653	0.14807	-0.00708	0.1095*	0.42 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0426 (2)	0.0565 (3)	0.0445 (2)	-0.0166 (2)	0.0092 (2)	-0.0128 (2)

S2	0.0545 (3)	0.0578 (3)	0.0449 (2)	-0.0180 (2)	0.0202 (2)	-0.0016 (2)
O1	0.0636 (8)	0.0544 (7)	0.0476 (7)	-0.0196 (6)	0.0227 (6)	0.0006 (5)
N1	0.0328 (6)	0.0310 (5)	0.0331 (6)	-0.0024 (5)	0.0088 (5)	-0.0023 (5)
C1	0.0341 (7)	0.0342 (7)	0.0343 (7)	-0.0077 (6)	0.0120 (6)	-0.0027 (6)
C2	0.0433 (8)	0.0364 (8)	0.0482 (9)	-0.0029 (6)	0.0166 (7)	-0.0041 (7)
C3	0.0592 (10)	0.0442 (9)	0.0529 (10)	-0.0167 (8)	0.0253 (8)	-0.0169 (8)
C4	0.0521 (10)	0.0673 (11)	0.0375 (8)	-0.0261 (9)	0.0150 (7)	-0.0131 (8)
C5	0.0379 (8)	0.0627 (10)	0.0371 (8)	-0.0096 (7)	0.0094 (6)	0.0066 (7)
C6	0.0385 (8)	0.0399 (8)	0.0395 (8)	-0.0039 (6)	0.0119 (6)	0.0011 (6)
C7	0.0395 (8)	0.0343 (7)	0.0359 (7)	0.0028 (6)	0.0118 (6)	0.0018 (6)
C8	0.0388 (8)	0.0481 (9)	0.0351 (7)	0.0015 (7)	0.0087 (6)	-0.0026 (7)
C9	0.0314 (7)	0.0319 (7)	0.0389 (7)	-0.0006 (5)	0.0091 (6)	-0.0025 (6)
C10	0.0505 (11)	0.0965 (17)	0.0611 (12)	-0.0013 (11)	-0.0027 (9)	0.0161 (11)

Geometric parameters (Å, °)

S1—C8	1.7952 (17)	C7—C8	1.496 (2)
S1—C9	1.7258 (15)	C2—H2	0.9300
S2—C9	1.6335 (15)	C3—H3	0.9300
O1—C7	1.199 (2)	C4—H4	0.9300
N1—C1	1.4406 (18)	C6—H6	0.9300
N1—C7	1.3943 (18)	C8—H8A	0.9700
N1—C9	1.3707 (18)	C8—H8B	0.9700
C1—C2	1.3814 (19)	C10—H10A	0.9600
C1—C6	1.376 (2)	C10—H10B	0.9600
C2—C3	1.381 (2)	C10—H10C	0.9600
C3—C4	1.371 (2)	C10—H10D	0.9600
C4—C5	1.384 (3)	C10—H10E	0.9600
C5—C6	1.392 (2)	C10—H10F	0.9600
C5—C10	1.503 (3)		
C8—S1—C9	93.63 (7)	C2—C3—H3	120.00
C1—N1—C7	120.71 (12)	C4—C3—H3	120.00
C1—N1—C9	122.13 (11)	C3—C4—H4	119.00
C7—N1—C9	116.98 (11)	C5—C4—H4	119.00
N1—C1—C2	119.55 (13)	C1—C6—H6	120.00
N1—C1—C6	118.46 (13)	C5—C6—H6	120.00
C2—C1—C6	121.98 (14)	S1—C8—H8A	110.00
C1—C2—C3	117.92 (15)	S1—C8—H8B	110.00
C2—C3—C4	120.60 (17)	C7—C8—H8A	110.00
C3—C4—C5	121.72 (16)	C7—C8—H8B	110.00
C4—C5—C6	117.93 (16)	H8A—C8—H8B	109.00
C4—C5—C10	122.24 (16)	C5—C10—H10A	109.00
C6—C5—C10	119.83 (17)	C5—C10—H10B	109.00
C1—C6—C5	119.84 (16)	C5—C10—H10C	109.00
O1—C7—N1	123.17 (14)	C5—C10—H10D	109.00
O1—C7—C8	125.44 (14)	C5—C10—H10E	109.00
N1—C7—C8	111.39 (12)	C5—C10—H10F	109.00

S1—C8—C7	106.86 (10)	H10A—C10—H10B	109.00
S1—C9—S2	122.64 (8)	H10A—C10—H10C	109.00
S1—C9—N1	111.07 (10)	H10B—C10—H10C	109.00
S2—C9—N1	126.29 (11)	H10D—C10—H10E	109.00
C1—C2—H2	121.00	H10D—C10—H10F	109.00
C3—C2—H2	121.00	H10E—C10—H10F	109.00
C9—S1—C8—C7	-2.42 (12)	C7—N1—C9—S2	179.24 (11)
C8—S1—C9—S2	-177.72 (10)	N1—C1—C2—C3	179.72 (14)
C8—S1—C9—N1	1.42 (11)	C6—C1—C2—C3	1.1 (2)
C7—N1—C1—C2	-85.14 (18)	N1—C1—C6—C5	179.89 (14)
C7—N1—C1—C6	93.56 (16)	C2—C1—C6—C5	-1.5 (2)
C9—N1—C1—C2	99.88 (17)	C1—C2—C3—C4	-0.2 (2)
C9—N1—C1—C6	-81.42 (18)	C2—C3—C4—C5	-0.4 (3)
C1—N1—C7—O1	3.2 (2)	C3—C4—C5—C6	0.0 (3)
C1—N1—C7—C8	-177.29 (12)	C3—C4—C5—C10	179.27 (17)
C9—N1—C7—O1	178.41 (14)	C4—C5—C6—C1	0.9 (2)
C9—N1—C7—C8	-2.06 (18)	C10—C5—C6—C1	-178.41 (15)
C1—N1—C9—S1	175.30 (10)	O1—C7—C8—S1	-177.61 (13)
C1—N1—C9—S2	-5.60 (19)	N1—C7—C8—S1	2.88 (16)
C7—N1—C9—S1	0.14 (16)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C8—H8B \cdots Cg2 ⁱ	0.97	2.59	3.5219 (17)	162
C7—O1 \cdots Cg1 ⁱ	1.20 (1)	2.94 (1)	4.1070 (16)	164 (1)

Symmetry code: (i) $-x+1, y+1/2, -z+1/2$.